



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	Art Unit: 1765
)	
Ho-Yong LEE et al)	Examiner: M. Anderson
)	
Appln. No.: 09/857,774)	Washington, D.C.
)	
Date Filed: June 11, 2001)	Confirmation No. 9764
)	
For: PEROVSKITE CRYSTAL)	ATTY.'S DOCKET:

DECLARATION UNDER 37 CFR 1.132

I, Ho-Yong Lee, hereby solemnly declare as follows:

I am the same Ho-Yong Lee who is a co-inventor and first named applicant of the above-identified U.S. patent application which relates to perovskite oxide crystals. My *curriculum vitae*, which demonstrates that I am an expert in the field of grain growth in polycrystalline materials, single crystal growth by grain growth, and perovskite oxide crystals, is attached and is made a part hereof.

An important problem which existed prior to our invention of the above-identified application was the high cost of large perovskite oxide crystals, as pointed out in the paragraph at the bottom of page 2 of our above-identified patent application. Our invention as disclosed in the above-identified U.S. patent application involves the "control" of abnormal grain growths, i.e., inducing the abnormal grain growths at the interface between a seed single crystal and a polycrystal, and repressing the abnormal grain growths inside the polysrystal.

As background, there are two broad categories of grain growth, namely "normal grain growth" and "abnormal grain growth".

Normal grain growth occurs when the entire growing grains grow at the same or approximately the same rate, whereby the resultant size of the grown grains increases while the size distribution of the entire grains remain constant. However, if some grains grow fast relative to other grains, and the resultant size distribution of the grains thus exhibits bimodal distribution, the grain growth is called abnormal grain growth. We have not discovered abnormal grain growth, but we have discovered how to control abnormal grain growth at the interface between the seed single crystal and the polycrystal and inside the polysrystal, thereby efficiently producing a large single crystal of perovskite oxide having the same composition as the polycrystal to which the seed single crystal was originally joined, as well as the same crystallographic structure as the seed single crystal.

Our claims have been rejected primarily on the basis of a U.S. patent in the name of Harmer and colleagues, U.S. 6,048,394 (hereinafter "the Harmer patent"). I have carefully studied the Harmer patent, and it does not at all mention abnormal grain growth, and it does not relate to the control of abnormal grain growth. It is a fact that the Harmer patent does not disclose what we do, how we do it, and the results we achieve, all as set forth in our above-identified U.S. patent application.

The Harmer patent does disclose forming a single crystal of relatively large size by bonding a seed crystal to a polycrystalline structure which the Harmer patent refers to as a "polycrystal precursor", and then heating. However, such a technique as disclosed in the Harmer is not able to produce consistent reliable results.

Basically, the Harmer patent is directed to a known method which is described in our patent application at the bottom of page 4, but this method is not suitable as there stated for "preparing single crystals large enough for practical uses... because the growth of single crystals is retarded...", i.e., too slow. And when abnormal grain growth appears, it is uncontrolled; i.e., as stated in the top paragraph on page 5, "it is difficult to produce single crystals having an actually applicable large size and the reproduction possibility is low because it is impossible to control the abnormal grain growths occurring inside the polycrystal..." This is the inevitable result achieved by following the Harmer patent, because the Harmer patent does not include any control of the abnormal grain growths occurring inside the polycrystal.

I repeat that control of the abnormal grain growth used in our invention is mandatory, and there are different ways of achieving such control, as disclosed in our above-identified U.S. patent application. One way is by a change of the ratio of the components or by an excess addition of specific components, which can be performed when a Pb-type perovskite polycrystal is prepared by the

Columbite precursor method, as stated in our claim 6 and the examples of our invention. In case of the excess addition of specific components, the excess amount should be such that abnormal grain growths are induced at the interface between a seed single crystal and a polycrystal, and repressed inside the polycrystal. Another way is by forming a temperature gradient such that the temperature of the single crystal side is high and the temperature of the polycrystal side is low, as stated in our claim 4.

As stated at the end of the second paragraph on page 6 of our above-identified U.S. patent application, success is achieved according to our invention by inducing abnormal grain growth at the interface between the polycrystal and the seed single crystal, but repressing abnormal grain growth inside the polycrystal. It is a fact that there is no hint of this in the Harmer patent.

A preferred embodiment of the Harmer patent involves the incorporation of a second ceramic, having a melting point lower than the main ferroelectric ceramic, between a seed crystal and the main ferroelectric ceramic. As explained at column 1, lines 48-55, of the Harmer patent, the second ceramic melts during annealing to form a liquid phase throughout the polycrystalline matrix and around the seed, thus providing a "wetting" effect for promotion of crystal growth.

However, as stated in the examples of our invention, our invention does not involve the incorporation of a second ceramic, having a melting point lower than the main ferroelectric ceramic,

between a seed crystal and the main ferroelectric ceramic that has already been prepared, but in some embodiments involves the excess addition of specific components in the amount greater than equivalent of the specific components in the composition of polycrystal when the polycrystal is prepared. To be more specific, as shown in Figure 5 and stated in example 3 of our invention, Mg is added in the excess amount (e.g., 1% Mg-extra), which is the amount greater than the chemical equivalent of Mg (e.g., 0% Mg-deficient) during the preparation of $(0.92) [(0.68) \text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3 - (0.32) \text{PbTiO}_3] - (0.08) \text{PbO}$. Namely, the specific components which are added in excess amount in our invention can never be the "second" ceramic of the Harmer patent.

Therefore, the means for promoting the growth velocity in the Harmer patent, i.e., the addition of the wetting second phase between a seed crystal and the main ferroelectric ceramic, does improve the mobility of the boundary between the seed crystal and the solid grains of the polycrystal. To the contrary, the means for promoting the growth velocity in the present invention, i.e., controlling (inducing and repressing) abnormal grain growth, "avoids" impingement between a growing single crystal seed and abnormal grains which appear in the polycrystal during the heat-treatment for single crystal growth and thus allows a single crystal seed to continue to grow into the polycrystalline body at a constant rate. In our invention, unlike the Harmer patent, there is no change in boundary mobility between the seed crystal and the solid grains of the polycrystal and no wetting effect is produced in our

invention which is the same as or similar to the wetting effect produced in the Harmer patent.

In accordance with our invention, we can grow the seed single crystal into the polycrystal at a constant growth velocity depending on the time of heat treatment, whereas the method of the Harmer patent cannot grow the seed single crystal at a constant growth velocity depending on the time of heat treatment. One can obtain a large single crystal having a size of several cm by using the present invention when the heat treatment continues for 50 hours or more. But not so in the method of the Harmer patent: as can be seen from Harmer's Fig. 10, even if heat treatment continues, the growth velocity jumps from nearly zero before formation of the wetting phase only during the formation of the wetting phase, and it is also nearly zero after volatilization of the wetting phase. Thus, even if the heat treatment in the method of the Harmer patent continues for 50 hours or more, one cannot obtain such a large single crystal as is obtained by our system.

I attach a table hereto which summarizes the important difference between our invention (the above-identified U.S. patent application) and the system of the Harmer patent.

	Present Invention	Harmer
Use and "control" of abnormal grain growth	Yes	Not mentioned
Inducing abnormal grain growth at the interface between a seed single crystal and a polycrystal, and repressing abnormal grain growth inside the polycrystal	Yes	Not mentioned
Whether grain boundary curvature is necessary	Not necessary	Necessary
Means for promoting the growth velocity	Control of abnormal grain growth	Addition of a wetting second phase between the seed and the polycrystal
Action of the means for promoting the growth velocity	Avoid impingement between a seed crystal and abnormal grains in the polycrystal and thus allow the seed crystal to grow continuously at a constant rate into the polycrystal	Increase in mobility between a seed crystal and solid grains in the polycrystal
The constancy of the growth velocity	The growth velocity is constant throughout heat treatment	The growth velocity largely varied with the time of heat treatment. The substantial growth occurs ONLY during a specific period and the growth velocity during most of the other periods is nearly ZERO.
Result of long-term heat treatment (50 hours or more)	Obtain a single crystal having a size of several cm	No matter how long the heat treatment is conducted, the size of the grown single crystal is limited to several mm (see Fig. 10)

In re of Appln. No. 09/857,774

I hereby further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

By Ho-Yong Lee
Ho-Yong Lee

Date: Feb. 17, 2004

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CURRICULUM VITAE

Name in Full: Lee, Ho-Yong (이 호 용; 李 壕 用)

Date of Birth: February 11, 1964

Present Position: Associate Professor,

Division of Materials and Chemical Engineering,

Sunmoon University

Address: Division of Materials and Chemical Engineering, Sunmoon University

100 Kalsanri, Tangjeongmyeon, Asan, Chungnam, 336-708, Korea

(Tel: 82-41-530-2366, 2306; Fax: 82-41-541-7426;

E-mail: hlee@sunmoon.ac.kr)

Major Field: Ceramic Processing, Microstructure Control, Grain Growth,
Solid-State Single Crystal Growth of Piezoelectric and Ferroelectric
Oxides (BaTiO₃, Ba(Ti,Zr)O₃, and PMN-PT, etc.)

Education:

B. S. in Mineralogy and Petroleum, Seoul National University, Korea

(1982. 3 ~ 1987. 2)

M. S. in Materials Science, Korea Advanced Institute of Science and

Technology, Korea

(1987. 3 ~ 1989. 2)

Thesis (M.S.) : Chemically Induced Interface Migration in Al₂O₃

(Advisor: Suk-Joong L. Kang)

Ph. D. in Materials Science, Korea Advanced Institute of Science and

Technology, Korea

(1989. 3 ~ 1993. 2)

Thesis (Ph.D.) : Coherency Strain Energy and Chemically Induced Grain

Boundary Migration in Al₂O₃ (Advisor: Suk-Joong L. Kang)

Experience:

Research Assistant, Korea Advanced Institute of Science and Technology

(1989. 3 ~ 1993. 2)

Instructor, Department of Materials Science and Engineering,

Sunmoon University

	(1993. 3 ~ 1994. 3)
Assistant Professor, Department of Materials Science and Engineering, Sunmoon University	
	(1994. 4 ~ 1999. 9)
Visiting Scholar, Materials Research Center, Lehigh University, PA, U.S.A.	
	(1995. 9 ~ 1997. 1)
Visiting Scholar, Dept. of Inorganic Materials, Tokyo Institute of Technology, Tokyo, Japan.	
	(1999. 1 ~ 1999. 2)
Associate Professor, Division of Metallurgical and Materials Engineering, Sunmoon University	
	(1999. 10 ~ present)
President, Ceracomp Co., Ltd.	
	(2000. 1 ~ present)

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- Centimeter-sized Piezoelectric Single Crystals (BaTiO_3 , $\text{Ba}(\text{Zr,Ti})\text{O}_3$ and PMN-PT) grown by the Solid-State Single Crystal Growth," 2002 U.S. Navy Workshop on Acoustic Transduction Materials and Devices, Baltimore, MD, U.S.A., May 13-15 (2002).
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